-29-.17. AU 1139 49404

EP 000590904 A1
APR 1994

94-111300/14 L03 NIHA 92.09.29
*EP 590904-A1
NIKKO KYODO CO LTD
92.12.07 92JP-351106 (+92JP-282297) *(94.04.06)* C23C 14/34
Sputtering target assembly with solid-phase diffusion bonded interfaces - has uniform crystal structure and can be manufactured to near-net geometry. (Eng)
R(DE GB IT)
C94-051341
Addnl. Data: OHHASHI T, FUKUYO H, SAWAMURA I, NAKAMURA K, FUKUSHIMA A, NAGASAWA M
93.09.27 93EP-307621; 92.11.24 92JP-334899
SR:2.Jnl.Ref EP342894 JP04143268 JP04143269

A sputtering target assembly comprises a target and a backing plate which are solid-phase diffusion bonded with or without inserts interposed between the two. The completed target assembly maintains the metallurgical characteristics and properties of the target prior to diffusion bonding. The target material has a melting point below 1000°C and has a uniform crystal structure with a grain size of 250 microns max..

Also claimed is a method of manufacturing the sputtering target described above where diffusion bonding is carried out under vacuum at 350-650°C..

USE

For use in semiconductor manufacture.

L(4-D2)

ADVANTAGE

The target can be manufactured to the final geometry or near net shape of the final geometry. The uniformity of the crystal structure is maintained. No deformation, degrading or unfavourable effects are imparted on the target material.

PREFERRED EMBODIMENTS

The target material is aluminium alloy, silver alloy, nickel alloy, W, Mo, Ti, Ta, Zr, or Nb. The inserts are silver or silver alloy.

The crystal grain size does not exceed 100 microns.(13 pp2010ACREDwgNo0/7).

EP-590904-A

(19) Eur päi ch s Patentamt
European Patent Office
Office europ´en des brevets

(11) Publication number : **0 590 904 A1**

(12) EUROPEAN PATENT APPLICATION

(21) Application number : **93307621.8**

(51) Int. Cl.^{5} : **C23C 14/34**

(22) Date of filing : **27.09.93**

(30) Priority : **29.09.92 JP 282297/92**
24.11.92 JP 334899/92
07.12.92 JP 351106/92

(43) Date of publication of application :
06.04.94 Bulletin 94/14

(84) Designated Contracting States :
DE GB IT

(71) Applicant : **NIKKO KYODO CO., LTD.**
10-1, Toranomon 2-chome
Minato-ku Tokyo (JP)

(72) Inventor : **Ohhashi, Tateo, c/o Nikko Kyodo Co., Ltd.**
Isohara Factory, 187-4 Usuba, Hanakawa-cho
Kitaibaraki-shi, Ibaraki-ken (JP)
Inventor : **Fukuyo, Hideaki, c/o Nikko Kyodo Co., Ltd.**
Isohara Factory, 187-4 Usuba, Hanakawa-cho
Kitaibaraki-shi, Ibaraki-ken (JP)
Inventor : **Sawamura, Ichiroh, c/o Nikko Kyodo Co., Ltd.**
Isohara Factory, 187-4 Usuba, Hanakawa-cho
Kitaibaraki-shi, Ibaraki-ken (JP)
Inventor : **Nakamura, Kenichirou, c/o Nikko Kyodo Co., Ltd.**
Isohara Factory, 187-4 Usuba, Hanakawa-cho
Kitaibaraki-shi, Ibaraki-ken (JP)
Inventor : **Fukushima, Atsushi, c/o Nikko Kyodo Co., Ltd.**
Isohara Factory, 187-4 Usuba, Hanakawa-cho
Kitaibaraki-shi, Ibaraki-ken (JP)
Inventor : **Nagasawa, Masaru, c/o Nikko Kyodo Co., Ltd.**
Isohara Factory, 187-4 Usuba, Hanakawa-cho
Kitaibaraki-shi, Ibaraki-ken (JP)

(74) Representative : **Drever, Ronald Fergus**
Swindell & Pearson 48, Friar Gate
Derby DE1 1GY (GB)

(54) Diffusion-bonded sputtering target assembly and method of manufacturing the same.

(57) A sputtering target assembly comprising a sputtering target and a backing plate characterized in that said sputtering target and backing plate is diffusion-bonded with or without an insert or inserts interposed there-between so as to have solid phase diffusion-bonded interfaces, said diffusion-bonded sputtering target substantially maintaining metallurgical characteristic and properties of the sputtering target before it is diffusion-bonded to said backing plate. The solid-diffusion bonding of the target and backing plate, with or without one or more insert interposed therebetween, at a low temperature and pressure, causes interdiffusion of their constituent atoms to attain high adhesion and bond strength without attendant deterioration or large deformation of the target material, while inhibiting the crystal growth in the target material. The bond thus obtained proves highly reliable because it undergoes no abrupt decrease in bond strength upon elevation of their service temperature and owing to the solid phase bonding, 100% bonding is achieved with noun-bonded portions such as pores left along the interfaces.

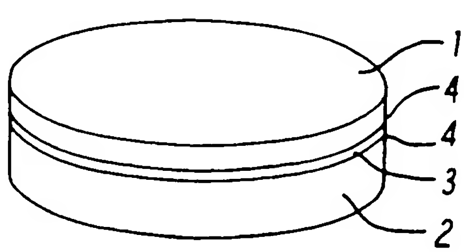

FIG. 1

EP 0 590 904 A1

under very great impact or heavy load such as a explosive bonding, hot press, HIP, or hot roll technique. This process causes serious deformation of th first metal member to be sputtered (target material), and attendant increased internal strains and the change of crystal structure.

Particularly, the uniformity as of crystal size and crystal orientation of a target is destroyed resulting in different crystal grain diameters and crystal orientations on various locations of the target. As a result, the quantity of sputter from the target begins to vary from point to point which leads to variation of deposited film thickness and hence deposited film properties. This problem is recently pointed out that this is a matter of serious concern. Further, the contamination of surface layer of the target produced is severe and so the yield of target material to be finished to the final size is very poor. Although it is also stated in the above mentioned publication that the first and second metal members may be bonded by explosive bonding after they have been machined to desired configurations, in that case, deformation of the target material and attendant increased internal strains and the change of crystal structure, and surface layer contamination are inevitable as stated above.

Recently, target materials having melting points below 1000° C, e.g., aluminum or aluminum alloys, have rapidly come into use for the wirings or interconnections of semiconductor devices. These target materials in many cases are supplied as finished to final geometry with very high purity. Such relatively lower melting target materials are susceptible to larger damages of its crystal structure, sometimes accompanied with coarsening of grain size of the target material.

[Object of the invention]

The present invention has for its object the development of a technique for bonding a target material finished to the final geometry or near net shape of the final geometry to a backing plate with a high strength while maintaining the uniformity of the crystal structure and imparting no deformative, degrading, or other unfavorable effect upon the target material itself.

[Summary of the invention]

The present inventors have searched for a bonding method for target materials which inhibits the crystal characteristics such as crystal grain growth and causes little deformative or other adverse effects upon the material. As a result, it has now been found that solid-phase diffusion bonding with or without the use of an insert produces a far better bond than expected in their interfaces. The diffusion bonding, performed while maintaining a solid phase under a light load (a low strain rate) in a vacuum, gives high adhesion and high bond strength with no or very small deformation of the target mat rial and with no un-bonded portions such as pores along the interfaces, whil inhibiting the destruction of uniform crystal structur, the growth of grains, etc. which the target material had before the bonding.

The term "solid-phase diffusion bonding" as used herein means a technique of bonding a target material and a backing plate with or without an insert or inserts sandwiched therebetween by diffusion along the interfaces under light heating and pressing conditions, whereby the two members are bonded while maintaining the solid phase rather than being melted, without causing unfavorable effects upon the target material including its grain growth and structure change.

Based upon this discovery, this invention provides a sputtering target assembly comprising a sputtering target and a backing plate characterized in that said sputtering target and backing plate are solid-phase diffusion-bonded with or without an insert or inserts interposed therebetween so as to have solid phase diffusion-bonded interfaces therebetween, said diffusion-bonded sputtering target substantially maintaining metallurgical characteristics and properties that the sputtering target had before it is diffusion-bonded to said backing plate.

It is convenient in explanation to divide target materials into ones having melting temperatures below and no less than 1000 °C and separately discuss them.

This invention, in its first aspect, provides:

(1-1) a solid-phase diffusion-bonded sputtering target assembly characterized by being composed of a target material having a melting point below 1000° C, one or more insert, and a backing plate, said target material, said insert and said backing plate having solid-phase diffusion bonded interfaces formed therebetween, said target material having uniform crystal structural with a grain size not exceeding 250 μm; and

(1-2) a method of manufacturing a sputtering target assembly, said target material having a grain size not exceeding 250 μm characterized by solid-phase diffusion bonding of a target material of a given final shape having a melting point below 1000 °C and a backing plate of a given final shape, with one or more inserts interposed therebetween, under a vacuum at a temperature between 150 and 300 °C.

Typical of the target material consists of aluminum or an aluminum alloy. The insert typically consists of silver or a silver alloy, copper or copper alloy, or nickel or a nickel alloy.

This invention, in a second aspect, provid s:

(2-1) a solid-phase diffusion-bonded sputt ring target assembly characterized by being composed of a target material having a melting point

°C and target materials having melting points of beyond 1000°C.

Typical examples of target materials having a melting point no more than 1000 °C are aluminum and aluminum alloys such as Al-Si-Cu, Al-Si, and Al-Cu alloys. Other alloy targets composed principally of such metals as Cu or Au also come within the contemplation of this group. As for insert materials, Ag, Cu, Ni, or their alloys are usually used. One or more such insert materials may be used in layers.

Examples of target materials having a melting point above 1000°C are target materials of refractory metals and their alloys, such as W, Mo, Ti, Ta, Zr, Nb, and W-Ti, and of high-melting compounds, such as high-melting silicides ($MoSi_x$, WSi_x, etc.). The material to be used as an insert herein is one or more of metals or alloys having a melting point lower than that of the target material. Typical of insert materials is Ag, Cu, Ni, or their alloy. For solid-phase diffusion bonding, the use of an insert material having a lower melting point than the target material employed is essential.

In the combination of a titanium target material and a titanium backing plate, the solid-phase diffusion-bonding is permitted with no use of an insert. As the titanium target materials, high-purity titanium target materials having a purity of 99.99% or upward are preferable. Titanium backing plates may be of ordinary industrial purity. For the purposes of the invention the term "titanium" is used to encompass the alloys with small percentages, up to 10% by weight, of alloying additives, such as Al, V, and Sn.

In fabricating a sputtering target assembly with the use of insert(s), a backing plate and a target material are degreased and rinsed with an organic solvent like acetone. Then, between the two is interposed an insert of one or more materials chosen from among Ag, Cu, Ni, and their alloys, desirably having at least 10μm thickness. The insert too must be degreased and rinsed beforehand. The use of a 10 μm or thicker insert is desirable because the micropores that result from surface irregularities, on the order of several micrometers, caused by machining of the surfaces of the target and backing plate to be bonded, would otherwise lessen the adhesive strength. The upper limit of thickness of the insert is not specified provided the insert is thick enough for solid-phase diffusion bonding. Excessive thickness is wasteful, however. A conventional foil, thin sheet or the like may be employed. For the material of the insert, Ag, Cu, Ni, or their alloy is suitable as referred to above, by reason of moderately high melting point and diffusion-ability to permit solid phase diffusion bonding. The insert is not limited to a single layer. Two or more superposed layers may be used instead. The surfaces to be bonded should be free from oxides or other impurities.

In the case of a target materials having a melting temperature no more than 1000 °C, a laminate consisting of a target material, a backing plate, and an insert is generally diffusion-bonded in a solid stat by holding it at a constant temperature within a b nding temperature range of 150-300°C, preferably of 150-250 °C, under a vacuum of 0.1 Torr or below and at a pressure of 1.0-20 kg/mm², preferably 3-10 kg/cm². In this way a sputtering target assembly is obtained. To avoid the formation of oxides, the bonding desirably is carried out in a vacuum atmosphere of 0.1 Torr or below. The choice of load to be applied depends upon the bonding temperature and the materials to be used. For sufficient pressure bonding to produce interfacial diffusion, the load must be at least 1.0 kg/mm². On the other hand, a load in excess of 20 kg/mm² can damage the target material. The bonding temperature is set within 150-300 °C for the following reasons. If it is below 150 °C insufficient diffusion of atoms results in poor adhesion. If it exceeds 300 °C crystal grain growth takes place in the target material. Moreover, because of the difference in thermal expansion rate, the target material and backing plate tend to warp or distort, leading to inadequate bonding.

In the case of target materials having melting points more than 1000° C, a laminate consisting of a target material, a backing plate, and an insert is generally diffusion-bonded in a solid state by holding it at a constant temperature within a bonding temperature range of 200-600 °C under a vacuum of 0.1 Torr or below and at a pressure of 0.1-20 kg/mm², preferably 3-10 kg/mm². In this way a sputtering target assembly is obtained. It is to avoid the formation of oxides that the bonding is carried out in a vacuum atmosphere of 0.1 Torr or below. The choice of the applicable load depends on the bonding temperature and the materials to be used. For sufficient pressure bonding to produce interfacial diffusion, the load must be at least 0.1 kg/mm². On the other hand, a load in excess of 20 kg/mm² can damage the target material. The bonding temperature is set within 200-600 °C for the following reasons. If it is below 200 °C insufficient diffusion of atoms results in poor adhesion. If it exceeds 600 °C the crystal structure, mechanical properties and the like of the target material and/or backing plate can deteriorate. Moreover, because of the difference in thermal expansion rate, the target material and backing plate tend to warp or distort, leading to inadequate bonding.

In the case where a titanium target material and a titanium backing plate are used, a laminate consisting of a target material and a backing plate is generally diffusion-bonded in a solid state by holding it at a constant temperature within a bonding temperature range of 350-650 °C, preferably of 450-600 °C, under a vacuum of 0.1 Torr or below and a load of 0.1-20 kg/mm², at a strain rate of $1x\ 10^{-3}$ /sec or below, preferably 1×10^{-4} / sec or below. In this way a sputtering target assembly is obtained. To avoid the formation of oxides, the bonding desirably is carried out in a va-

of the bond strength values under shear of these bonded materials was similar as in FIG. 3. The bond strength und r shear of the laminate using th Sn-Pb-Ag low-melting brazing material is about 3 kg/mm², while the material solid-phase diffusion-bonded in accordance with this invention has about twice the strength, the values being around 6 kg/mm². As for the temperature dependence, the bond strength under shear of the material using the Sn-Pb-Ag low-melting brazing material becomes zero in the vicinity of 180 °C which is the melting point of the brazing material itself. The solid-phase diffusion-bonded material of this invention, by contrast, exhibits a bond strength under shear of 3 kg/mm² or more above 200 °C and retains a strength of 2 kg/mm² even at 250 °C.

(Example 4)

A tungsten target material of high purity (>99.999%) in the form of a disk 295 mm in diameter was diffusion-bonded to a titanium backing plate of industrial purity through an Ag insert in a vacuum of 5 x 10^{-6} Torr, at a bonding temperature of 400° C, and under a load of 8 kg/mm². A micrograph of a cross section illustrating the bond interfaces of the bonded material thus obtained is shown in FIG. 5. It can be seen from the photograph that interfaces having the bonded area percentage of 100% with non-bonded portions such as pores were obtained. The bond strength under shear at room temperature of test pieces cut out from five diametral points in the manner described in Example 3 was 7 kg/mm². On the other hand, the bond strength under shear of test pieces of a material bonded using an In brazing material was at a level of as lower as of 1 kg/mm². This difference verifies the superiority of solid-phase diffusion bonding.

(Example 5)

Targets were made by solid-phase diffusion bonding similarly to Example 3 but using inserts of copper foil or nickel foil. Similar effects were attained.

(Example 6)

A high-purity (>99.999%) titanium target in the form of a disk 295 mm in diameter was diffusion-bonded to a titanium backing plate of industrial purity directly without the use of an insert under a vacuum of 5 x 10^{-6} Torr and at a bonding temperature of 550 °C, load of 7.5 kg/mm², and strain rate of 2x 10^{-5}/sec. In FIG. 6 are compared the bond strength under shear at room temperature of an assembly made by solid-phase diffusion bonding in accordance with this invention with that of a assembly which used an In brazing material. A micrograph of the bond interface of the bonded assembly is shown in FIG. 7. The crystal grain size of the target after bonding was 50 μm. The photograph clearly indicates that the interface had attained 100% bonding without non-bonded portions such as pores. Th test piece at room t mp rature xhibited a bond strength under sh ar of 25 kg/mm² and a tensile strength under shear of 43 kg/mm². The In brazing material-bonded piece gave a bond strength under shear at a low level of 1 kg/mm². This testifies to the superiority of solid-phase diffusion bonding.

(Example 7)

A target assembly was made by solid-phase diffusion bonding in the same manner as described in Example 6 with the exception that the bonding temperature was changed to 500 °C and the strain rate to 1 x 10^{-5}/sec. Similar effects were achieved.

[Advantages of the invention]

Solid-phase diffusion bonding at a low temperature and pressure has the following features:

(1) The uniformity of crystal structure is maintained with the suppression of crystal grain growth.

(2) The process of fabrication causes no damage to the target material.

(3) Interdiffusion of the atoms constituting the target material, backing plate, and insert if used across the bond interfaces produces high degrees of adhesion and bond strength.

(4) The sharp drop of bond strength is avoided as found in the rise of the service temperature that can occur with a low-melting brazing material.

(5) Solid-phase bonding gives reliable bonds of a bonding area percentage of 100% without non-bonded portions such as pores that can result from ordinary bonding, due to shrinkage on solidification of a brazing material.

Consequently, this invention offers advantages as follows:

(a) A target material can be bonded to a backing plate without the possible danger of being damaged ; (b) uniformity of sputtering is ensured with the result that the film thickness is kept constant and the film properties are made uniform and stable; (c) a greater electric power can be put for sputtering, and therefore the throughput for film forming by sputtering can be improved; and (d) the target itself can be baked at around 200 °C, thus reducing adsorbed water, gas, and the like in the target surface.

Claims

1) A sputtering target assembly comprising a sputtering target and a backing plate characterized in that said sputtering target and backing plat ar solid-phase diffusion-bonded with or without an insert or

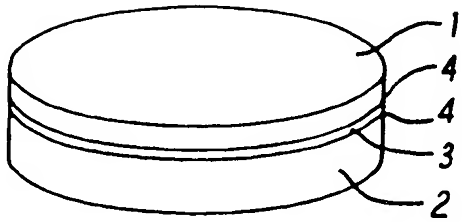
1
4
4
3
2

FIG. 1

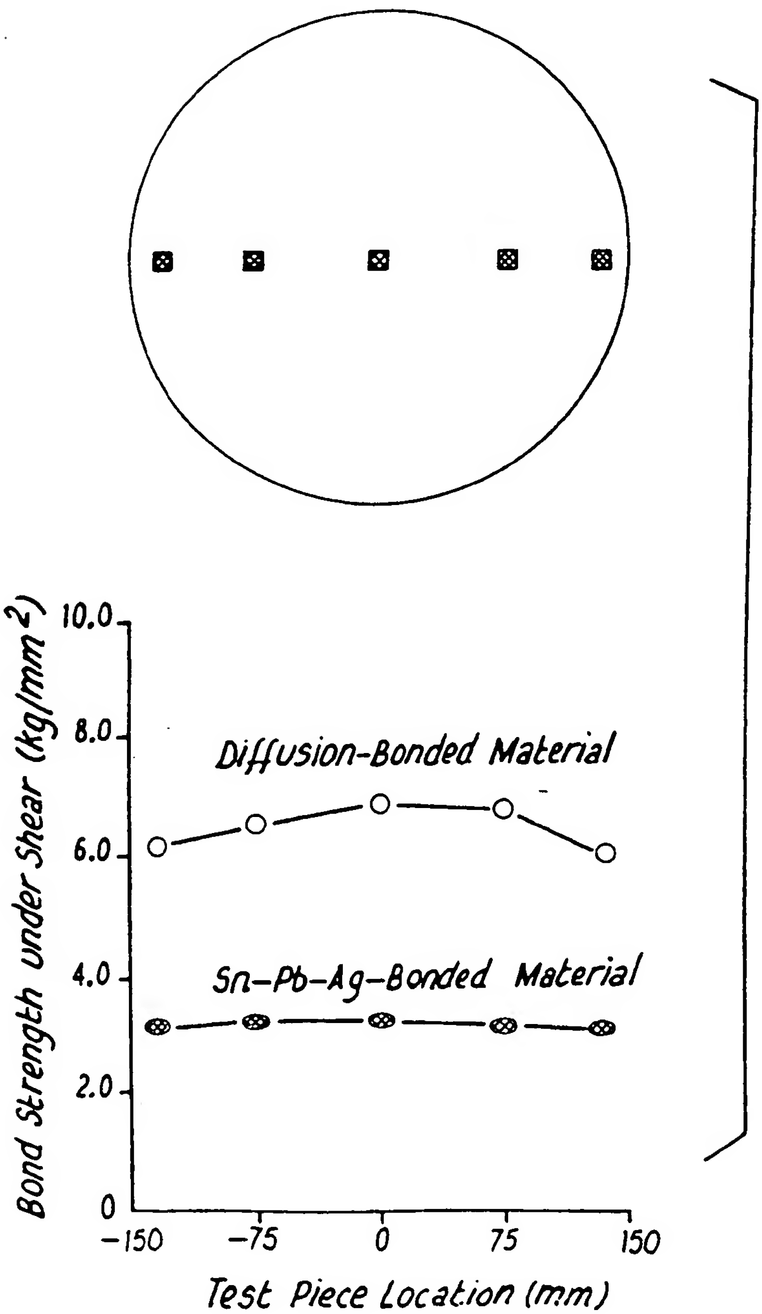
Bond Strength under Shear (kg/mm²)
10.0
8.0
6.0
4.0
2.0
0
Diffusion-Bonded Material
Sn-Pb-Ag-Bonded Material
-150
-75
0
75
150
Test Piece Location (mm)

FIG. 2

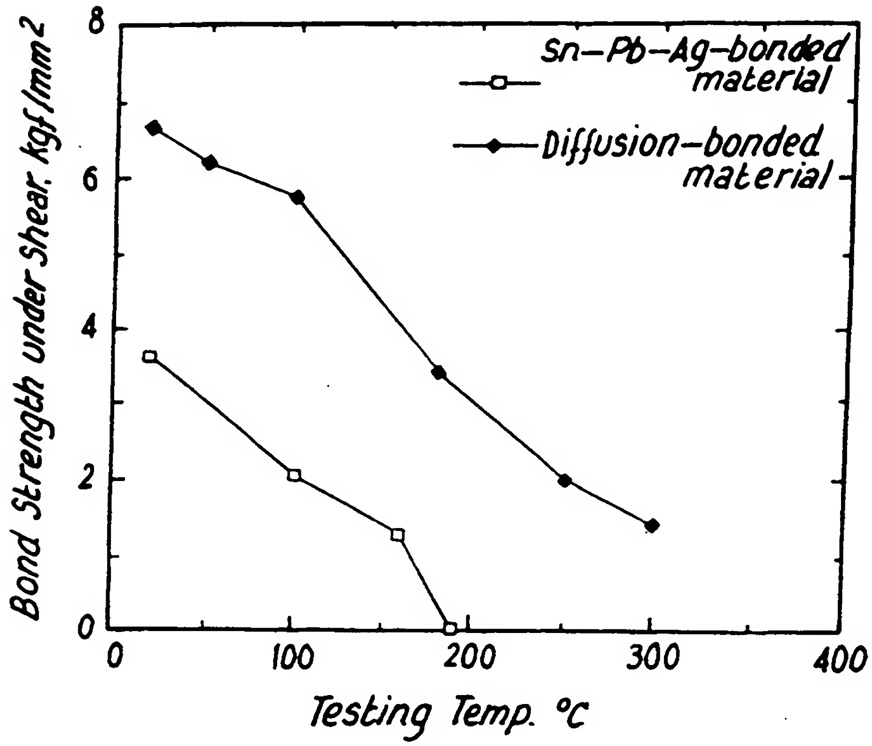
Bond Strength under Shear, kgf/mm2
8
6
4
2
0
0
100
200
300
400
Testing Temp. °C
Sn–Pb–Ag–bonded material
Diffusion–bonded material

FIG. 3

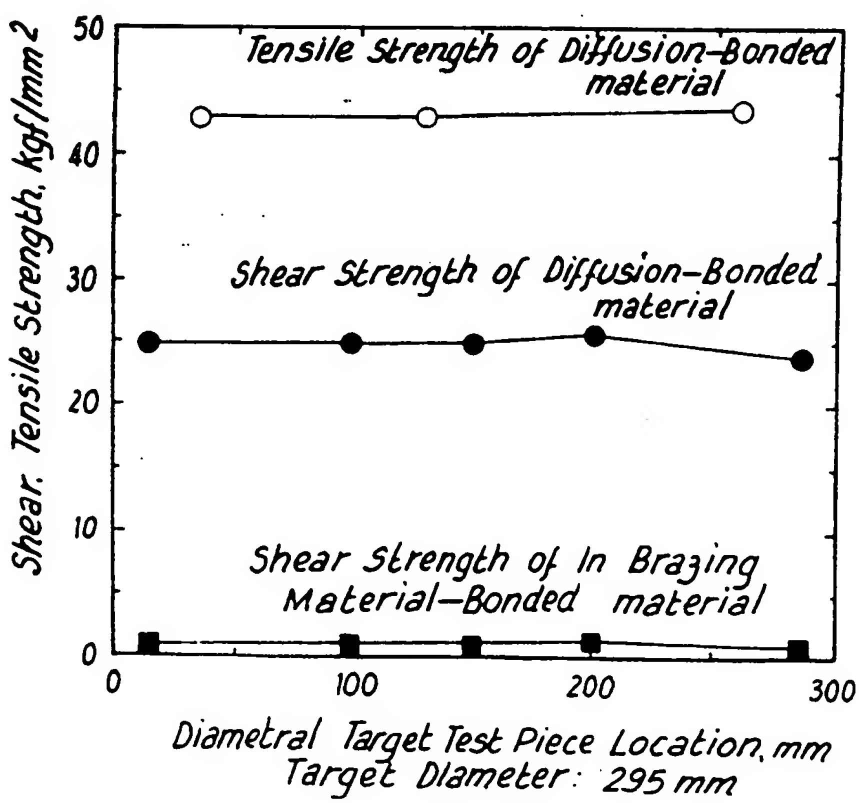
Shear. Tensile Strength, kgf/mm2
50
40
30
20
10
0
0
100
200
300
Diametral Target Test Piece Location, mm
Target Diameter: 295 mm
Tensile Strength of Diffusion–Bonded material
Shear Strength of Diffusion–Bonded material
Shear Strength of In Brazing Material–Bonded material

FIG. 6

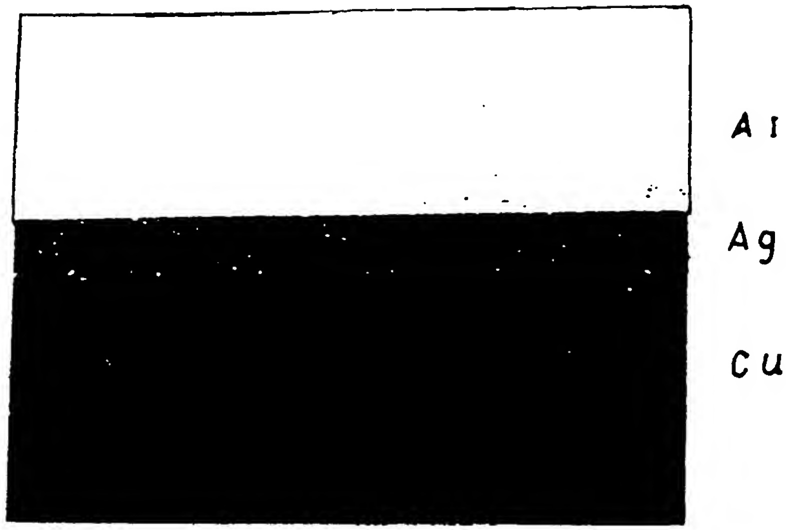
A l
Ag
Cu

FIG. 4

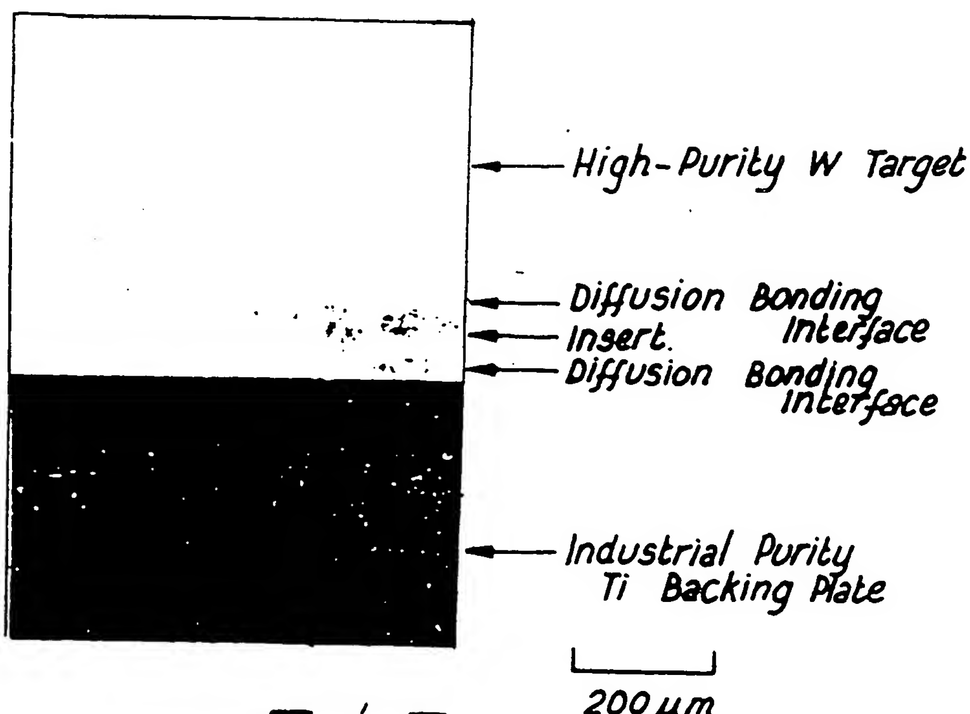
High-Purity W Target
Diffusion Bonding Interface
Insert.
Diffusion Bonding Interface
Industrial Purity Ti Backing Plate
200 μm

FIG. 5

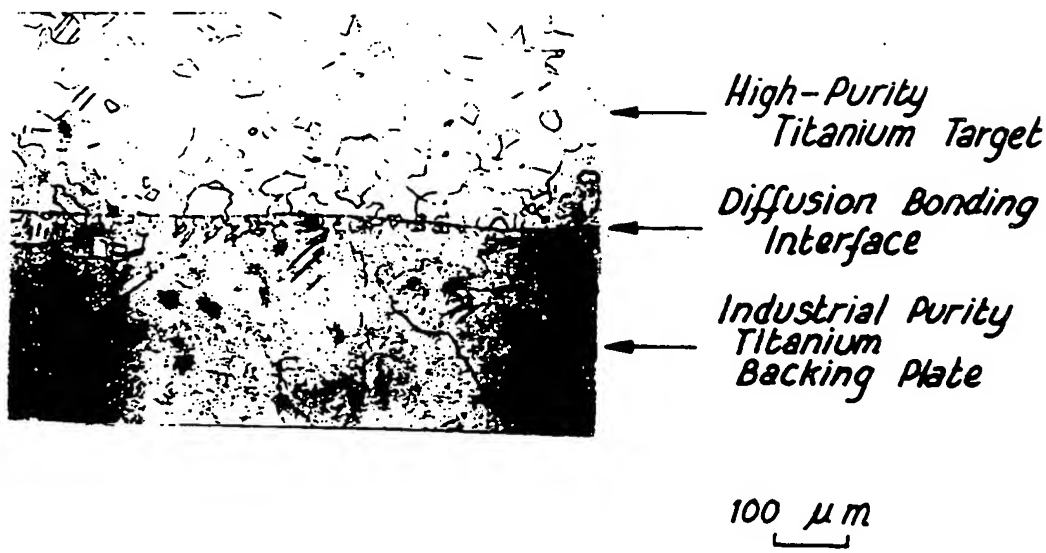
High-Purity
Titanium Target
Diffusion Bonding
Interface
Industrial Purity
Titanium
Backing Plate
100 μm

FIG. 7

European Patent Office

EUROPEAN SEARCH REPORT

Application Number

EP 93307621.8

DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.5)
D,A	PATENT ABSTRACTS OF JAPAN, unexamined applications, C section, vol. 16, no. 419, September 03, 1992 THE PATENT OFFICE JAPANESE GOVERNMENT page 38 C 981 * No. 04-143 268 (FUJITSU LTD) *	1-14	C 23 C 14/34
D,A	PATENT ABSTRACTS OF JAPAN, unexamined applications, C section, vol. 16, no. 419, September 03, 1992 THE PATENT OFFICE JAPANESE GOVERNMENT page 38 C 981 * No. 04-143 269 (FUJITSU LTD) *	1-14	
A	EP - A - 0 342 894 (KABUSHIKI KAISHA TOSHIBA) * Claims *	1-14	TECHNICAL FIELDS SEARCHED (Int. Cl.5) C 23 C 14/00

The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
VIENNA	03-01-1994	HOFBAUER

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
Y : particularly relevant if combined with another document of the same category
A : technological background
O : non-written disclosure
P : intermediate document

T : theory or principle underlying the invention
E : earlier patent document, but published on, or after the filing date
D : document cited in the application
L : document cited for other reasons

& : member of the same patent family, corresponding document